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(54) **LUBRICATING AGENT FOR MOLD AT ELEVATED TEMPERATURE, IRON-BASED POWDER COMPOSITION FOR ELEVATED TEMPERATURE COMPACTION WITH LUBRICATED MOLD AND HIGH DENSITY FORMED PRODUCT FROM IRON-BASED POWDER COMPOSITION, AND METHOD FOR PRODUCING HIGH DENSITY IRON-BASED SINTERED COMPACT**

(57) A process for producing a high-density iron-based green compact is provided which can form a green compact with a high density. Also provided is a process for producing a sintered compact from the green compact. A specified combination lubricant is applied to the surface of a die for pressure compaction by electrical charging, which lubricant is composed of a lubricant having a higher melting point than a preset compaction temperature, and a lubricant having a lower melting point than the compaction temperature. A heated iron-based powder mixture is filled into the die, fol-

lowed by pressure compaction, whereby a green compact is formed. The green compact can be sintered to provide a sintered compact. The powder mixture comprises an iron-based powder, a powder compaction lubricant and a graphite powder, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than the compaction temperature and in a content from 10 to 75% by mass, and a lubricant having a higher melting point than the compaction temperature and as the balance, and the content of the graphite powder is less than 0.5% by mass based on the total amount of the iron-based powder mixture.

Description**BACKGROUND OF THE INVENTION**5 **1. Field of the Invention**

[0001] This invention relates to processes for the production of iron-based powder green compacts and iron-based sintered compacts for powder metallurgy. More particularly, the invention relates to improvements in lubricants for use in producing a high-density, green compact made from iron-based powder by warm compaction.

10 **2. Description of the Related Art**

[0002] In general, a powdered iron-based green compact for powder metallurgy is produced by filling an iron-based powder mixture into a die. The powder mixture is generally derived by mixing an iron-based powder with alloying powders such as copper powder, graphite powder and the like and further with lubricants such as zinc stearate, lead stearate and the like, and then by subjecting the iron-based powder mixture to. The resultant green compact usually has a density in the range from 6.6 to 7.1 Mg/m³.

[0003] Such a green compact is further sintered to obtain a sintered compact which, where desired, is sized or cut into a powder metallurgical product. Where great strength is required, carburizing heat treatment or brightening heat treatment is in some instances performed after completion of the sintering.

[0004] The above powder metallurgy permits components parts of complicated shapes to be formed with high dimensional accuracy and in near net structure, significantly saving the cost of cutting work as contrasted to conventional production methods.

[0005] With regard to powder metallurgical iron products, a keen demand has recently been made for more higher dimensional accuracy to omit cutting work and to save production cost, and also for more greater strength to make components parts small in size and light in weight.

[0006] In order to give greater strength to a powder metallurgical product (a sintered compact), it is beneficial to form high-density sintered compacts from an iron-based green compact which has been produced to have a high density. As the density of a sintered compact increases, the number of voids in the compact decreases so that the component part is obtainable with improved mechanical properties such as tensile strength, impact value, fatigue strength and the like.

[0007] As warm compaction techniques evolved to form a high-density iron-based green compact, there have been proposed a double molding-double sintering method in which an iron-based powder mixture is pressed and sintered in the usual manner, followed by repeated pressing and sintering, and a sinter forging method in which single pressing and single sintering are performed, followed by hot forging.

[0008] Moreover, warm compaction techniques are known in which metal powder is compacted with heat as disclosed for instance in Japanese Unexamined Patent Application Publication No. 2-156002, Japanese Examined Patent Application Publication No. 7-103404, U.S. Patent No. 5,256,185 and U.S. Patent No. 5,368,630. Such a warm compaction techniques are designed to melt and disperse a lubricant partly or wholly between the metallic particles, thereby reducing the frictional resistance between the metallic particles and the frictional resistance between the green compact and an associated die, so that improved compressibility is attained. The compaction technique noted here is thought to be most advantageous in view of possible cost savings amongst the methods previously mentioned for the production of high-density iron-based green compacts. A green compact of about 7.30 mg/m³ in density can be obtained by the above compaction technique when an iron-based powder mixture is compacted at a pressure of 7 t/cm² and at a temperature of 150 C, which powder mixture is derived by mixing a partially alloyed iron powder of a Fe-4Ni- 0.5Mo-1.5Cu with 0.5% by mass of graphite and 0.6% by mass of lubricant.

[0009] However, according to the warm compaction techniques of the above-cited publications, i.e., Japanese Unexamined Patent Application Publication No. 2-156002, Japanese Examined Patent Application Publication No. 7-103404, U.S. Patent No. 5,256,185 and U.S. Patent No. 5,368,630, the problem arises that an iron-based powder mixture is less fluid and hence less productive, the resultant green compact is irregular in respect of densities, and the resultant sintered compact is unfavorably variable in respect of physical properties. Another drawback is that a high force must be applied to draw the green compact from the corresponding mold with consequent marred surface of the product and shortened lifetime of the die.

[0010] In these warm compaction techniques, a lubricant is also contained in an iron-based powder mixture so as to reduce the resistance between the metallic particles and the resistance between the green compact and the associated mold, thereby providing improved compressibility. During warm compaction, the lubricant is partly or wholly melted and then pushed to locate adjacent to the surface of the green compact. Upon subsequent sintering, the lubricant gets thermally decomposed or volatilized and hence escapes from the green compact, leaving coarse voids near to the

surface of the sintered compact. This poses the problem that the sintered compact results in insufficient mechanical strength.

[0011] To cope with this problem, Japanese Unexamined Patent Application Publication No. 8-100203 discloses that when room temperature compaction or warm compaction is effected, the content of a lubricant to be incorporated in an iron-based powder mixture is decreased by coating the surface of a die with an electrical charged lubricant powder such that a high-density green compact is produced. In this technique, however, the coating lubricant is susceptible to morphological changes at around its melting point since it is of a single nature so that the lubricating action is largely variable. This has the drawback that the compaction temperature range depends restrictedly upon the melting point of the coating lubricant. Also defectively, even if the content of the lubricant in the powder mixture can be decreased with the coating lubricant applied on to the mold surface, the content of the former lubricant may be too low which is dependent upon the lubricant components to be incorporated in the powder mixture. In this instance, the former lubricant does not exhibit lubrication, failing to enhance the density of a pressurized powder.

[0012] From the viewpoints of great strength and cost saving of automotive parts, there has been a need for the development of a process capable of producing an iron-based green compact with a higher density but by single compaction.

SUMMARY OF THE INVENTION

[0013] In order to eliminate the foregoing problems of the conventional art, a first object of the present invention is to provide a process for producing a high-density iron-based green compact which permits a high-density green compact to be formed with a density of 7.4 Mg/m^3 or above and by single pressing when warm pressure compaction is effected as to an iron-based powder mixture derived by mixing a partially alloyed iron powder of, for example, a Fe-4Ni-0.5Mo-1.5Cu composition, with 0.5% by mass of a graphite powder.

[0014] A second object of the invention is to provide a process for producing a high-density iron-based sintered compact which permits a high-density sintered compact to be formed by sintering such an iron-based green compact.

[0015] To achieve the above objects by utilizing a warm compaction technique and a die lubrication compaction technique, the present inventors have conducted intensive researches on various lubricants for mold lubrication and various formulations of iron-based powder mixtures containing lubricants. It has now been found that the force for drawing an iron-based green compact from the corresponding mold can be effectively lessened by the use of a certain specific combination lubricant as a lubricant for mold lubrication. This combination lubricant is composed in a suitable ratio of a lubricant having a lower melting point than a preset compaction temperature and a lubricant having a higher melting point than the compaction temperature and can be applied to the surface of a preheated die by electrical charging.

[0016] The present invention has been made on the basis of the aforesaid finding and further supporting studies.

[0017] More specifically, according to a first aspect of the present invention, there is provided a lubricant for warm mold lubrication, comprising a mixture of a lubricant having a higher melting point than a preset compaction temperature, and a lubricant having a lower melting point than the compaction temperature, the lubricant for mold lubrication being applicable to the surface of a preheated die by means of electrical charging when a powdered material is compacted in the mold by pressure compaction.

[0018] According to a second aspect of the invention, there is provided a die lubricant for warm compaction with die, comprising a lubricant having a higher melting point than a preset compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance, the lubricant being applicable to a surface of a preheated die by means of electrical charging when a powdered material is compacted in the mold by pressure compaction.

[0019] In this aspect, the higher-melting lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure.

[0020] In this aspect, the lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.

[0021] According to a second aspect of the invention, there is provided an iron-based powder mixture for warm compaction with die lubrication, comprising an iron-based powder and a powder compaction lubricant, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.

[0022] According to this aspect of the invention, there is provided an iron-based powder mixture for warm compaction with die lubrication, comprising an iron-based powder, a powder compaction lubricant and a graphite powder, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance, and the content of the

graphite powder is less than 0.5% by mass based on the total amount of the iron-based powder mixture.

[0023] In the second invention, the content of the powder compaction lubricant is in the range from 0.05 to 0.40% by mass.

[0024] According to the third invention, there is provided a process for the production of a high-density iron-based green compact, comprising the steps of: preheating a die at a selected temperature; applying a die lubricant for warm compaction with die to the surface of the mold by electrical charging; filling a heated iron-based powder mixture in the mold; and then subjecting the mixture to pressure compaction at a preset compaction temperature, wherein the die lubricant for warm compaction with die lubrication comprises a lubricant having a higher melting point than the compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance; and the iron-based powder mixture comprises an iron-based powder and a powder compaction lubricant, the powder compaction lubricant comprising a lubricant having a lower melting point than the compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.

[0025] In this third invention, the graphite powder can be also added in a content less than 0.5% by mass based on the total amount of the iron-based powder mixture.

[0026] In the third invention, the higher-melting lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a layer crystal structure.

[0027] The lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.

[0028] The lubricant for in the powder mixture is preferably added in a amount from 0.05 to 0.40% by mass.

[0029] The present invention can also provide a high-density sintered compact produced by single pressing.

[0030] In a fourth embodiment of the invention, there is provided a process for the production of a high-density iron-based sintered compact, comprising the step of further sintering the high-density iron-based green compact produced by the process according to any one of the fifth and sixth aspects, thereby forming a sintered compact.

[0031] The above and other objects, features and advantages of the present invention will become manifest upon reading of the following detailed description.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0032] In the practice of the present invention, a heated iron-based powder mixture is filled in a die and then molded by pressure compaction at a preset compaction temperature, whereby an iron-green compact is obtained.

[0033] In the invention, a die to be used is preheated at a suitable temperature. The preheating temperature is not particularly restricted so long as an iron-based powder mixture can be maintained at a preset compaction temperature. The preheating temperature is set to be preferably higher than the compaction temperature by 20 to 60 C.

[0034] An electrically charged lubricant for mold lubrication is introduced into a preheated die to apply the lubricant to the surface of the mold by electrical charging. Desirably, the lubricant (solid powder) for mold lubrication is placed in a die lubricating system (for example, Die Wall Lubricant System manufactured by Gasbarre Co.) where electrical charging is performed by means of contact charging between the solid lubricant particles and the inner wall of the system. The electrically charged lubricant is jetted into the mold and applied to the mold surface by electrical charging. The amount of the lubricant to be applied to the mold surface by electrical charging is set preferably in the range from 5 to 100 g/m². Amounts less than 5 g/m² result in insufficient lubricating action, calling for a high force to draw the resultant green compact from the mold. Amounts more than 100 g/m² cause the lubricant to remain on the product surface, making the product unsightly in appearance.

[0035] The die lubricant for warm compaction with die lubrication is used in electrically charged relation to the surface of a preheated die when a powdered material is compacted by pressure compaction. This lubricant is a mixture of a lubricant having a higher melting point than a preset compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance. The preset compaction temperature used herein denotes a temperature as measured on the mold surface at the time pressure compaction is carried out.

[0036] The higher-melting lubricant is present in a sold state in the die lubricant for warm compaction with die lubrication at the time compaction is effected, and it behaves like a solid lubricant that acts as "a roller" within a die, consequently lessening the force for drawing a green compact from the mold. Moreover, such higher-melting lubricant has a role to prevent a completely or partially molten lubricant (a lower-melting lubricant to be described later) from getting migrated within the mold, decreasing the frictional resistance between the green compact and the mold surface so that the force for product drawing is prevented from being unfavorably increased.

[0037] If the content of the higher-melting lubricant is less than 0.5% by mass, the lower-melting lubricant becomes

relatively abundant. This causes a large amount of a molten lubricant that migrates within a die and does not distribute uniformly on the surface of the mold, increasing the frictional resistance between the green compact and the mold surface and hence failing to lessen the force for product drawing to a sufficient extent. Conversely, if the content of the higher-melting lubricant is more than 80% by mass, a lubricant not subject to melting in a die is too large in amount for uniform distribution on the die surface. This is responsible for diminished mold lubrication and hence for increased force for product drawing. Hence, the content of the higher-melting lubricant present in the die lubricant for warm compaction with die lubrication should be within the range from 0.5 to 80% by mass.

[0038] The lubricant for mold lubrication contains, in addition to the above-specified higher-melting lubricant, a lubricant having a lower melting point than the preset compaction temperature. This lower-melting lubricant melts completely or partially at the compaction temperature and presents a grease-like state on the surface of a die, exerting a beneficial effect on lessening the force for drawing a green compact from the mold.

[0039] The higher-melting lubricant is preferably at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used.

[0040] As the metallic soap, zinc stearate, lithium stearate, lithium hydroxystearate or the like is preferred. As the thermoplastic resin, polystyrene, polyamide, fluorine resin or the like is preferred. As the thermoplastic elastomer, polystyrene elastomer, polyamide elastomer or the like is preferred. The inorganic lubricant of a lamellar crystal structure is graphite, MoS₂ or carbon fluoride, and finer particle sizes are more effective in lessening the force for product drawing. The organic lubricant of a lamellar crystal structure is melamine-cyanuric acid adduct (MCA) or N-alkyl aspartate- -alkyl ester.

[0041] Meanwhile, the lower-melting lubricant is desired to be a lubricant that melts completely or partially at the compaction temperature and tends to applied to the surface of a die at a low melting point by electrical charging. This lower-melting lubricant is preferably at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used. As the metallic soap, zinc stearate or calcium stearate is preferred. As the amide wax, ethylene bis-stearoamide, monoamide stearate or the like is preferred. As the eutectic mixture, ethylene bis-stearoamide-polyethylene eutectic, ethylene bis-stearoamide-zinc stearate eutectic, ethylene bis-stearoamide-calcium stearate eutectic is preferred.

[0042] Subsequently, a heated iron-based powder mixture is charged into a die electrically charged with a lubricant for mold lubrication, followed by pressure compaction, whereby a green compact is obtained.

[0043] The iron-based powder mixture is heated preferably at from 70 to 200 C. Lower temperatures than 70 C result in an iron powder having increased yield stress and hence lead to a green compact having decreased density. Inversely, higher temperatures than 200 C show no appreciable rise in density, arousing a fear of an iron powder getting oxidized. Thus, the temperature at which the iron-based powder mixture is heated should be set within the range from 70 to 200 C.

[0044] The iron-based powder mixture is derived by mixing an iron-based powder with a lubricant (a powder compaction lubricant) or an alloying powder. The method of mixing the iron-based powder with the compaction lubricant or the alloying powder is not particularly restrictive, but any known method is suitably useful. In the case where the iron-based powder is mixed with the alloying powder, it is desired that after completion of primary mixing in which the iron-based powder and alloying powder are mixed with a part of the powder compaction lubricant, secondary mixing be performed in which the resultant mixture is stirred with heat at a higher temperature than the melting point of at least one of the aforesaid lubricant in order to melt the one lubricant, and the mixture having been melted is cooled with stirring to thereby apply the one lubricant to the surface of the iron-based powder mixture so that the alloying powder is bonded, followed by mixing of the balance of the powder compaction lubricant.

[0045] The iron-based powder according to the present invention is selected from among pure iron powders such as an atomized iron powder, a reduced iron powder or the like, a partially diffusively alloyed steel powder, a completely alloyed steel powder, and a mixed powder thereof.

[0046] The content of the powder compaction lubricant in the iron-based powder mixture is set preferably in the range from 0.05 to 0.40% by mass based on the total amount of the iron-based powder mixture. Contents less than 0.05% by mass make the resultant iron-based powder mixture less fluid and fail to apply the lubricant uniformly to the surface of a die, producing a green compact having decreased density. Conversely, contents more than 0.40% by mass suffer high voiding after sintering and give a sintered compact having decreased density.

[0047] The powder compaction lubricant contained in the iron-based powder mixture is a mixed lubricant obtained by mixing a lubricant having a lower melting point than the preset compaction temperature and a lubricant having a higher melting point than the compaction temperature. The content of the lower-melting lubricant in the powder compaction lubricant is in the range from 10 to 75% by mass, whereas the content of the higher-melting lubricant is in the range from 25 to 90% by mass as the balance. The lower-melting lubricant is effective in that it melts during pressure compaction, penetrates in between the iron-based particles by capillary action, disperses uniformly in the particles, reduces particle-to-particle contact resistance and facilitates reorientation of iron-based particles, thus accelerating

the enhancement of product density. If the content of the lower-melting lubricant is less than 10% by mass, such lubricant fails to disperse uniformly in the iron-based particles and suffers poor density of a green compact. If the content of the lower-melting lubricant is more than 75% by mass, a molten lubricant is squeezed toward the surface of a die as the density of a green compact is increased so that passages are provided on the product surface for the molten lubricant to escape out of the product. The passages cause many coarse voids on the product surface, giving insufficient strength to the resultant sintered compact.

[0048] The higher-melting lubricant contained in the iron-based powder mixture is present in a solid state at the time compaction is effected. This lubricant acts as "a roller" on the surface protrusions of iron-based particles where it repels a molten lubricant, promoting particle reorientation and enhancing product density.

[0049] The higher-melting lubricant contained in the powder compaction lubricant for the iron-based powder mixture is preferably at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used.

[0050] As the metallic soap, zinc stearate, lithium stearate, lithium hydroxystearate or the like is preferred. As the thermoplastic resin, polystyrene, polyamide, fluorine resin or the like is preferred. As the thermoplastic elastomer, polyethylene elastomer, polyamide elastomer or the like is preferred. As the inorganic lubricant of a lamellar crystal structure, graphite, MoS₂ or carbon fluoride is preferred, and finer particle sizes are more effective for lessening the force for drawing a green compact from a die. As the organic lubricant of a lamellar crystal structure, melamine-cyanuric acid adduct (MCA) or N-alkyl aspartate- -alkyl ester is preferred.

[0051] The lower-melting lubricant contained in the powder compaction lubricant for the iron-based powder mixture is preferably at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof. Suitable examples are chosen from the following lubricants depending upon the compaction temperature used.

[0052] As the metallic soap, zinc stearate, calcium stearate or the like is preferred. As the amide wax, ethylene bis-stearoamide, monoamide stearate or the like is preferred. As the eutectic mixture, ethylene bis-stearoamide-polyethylene eutectic, ethylene bis-stearoamide-zinc stearate eutectic, ethylene bis-stearoamide-calcium stearate eutectic or the like is preferred. Though dependent upon the compaction temperature used, some of these lower-melting lubricants may be utilized as higher-melting lubricants.

[0053] Graphite can be used as an alloying powder in the iron-based powder mixture. This graphite powder is effective to reinforce a sintered compact to be produced, but too high a content is liable to decrease product density largely. Hence, the content of graphite should be set to be less than 0.5% by mass based on the total amount of the iron-based powder mixture.

[0054] In the present invention, the high-density iron-based green compact formed by the above-specified production process can be further sintered, whereby a high-density iron-based sintered compact is obtained. Here, any conventional sintering method may be suitably used without limitation. Sinter hardening may also be used by which rapid cooling is effected after sintering to enhance product strength.

[0055] The present invention may be more fully understood with reference to the following examples.

Example 1

[0056] A partially alloyed steel powder of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder was used as an iron-based powder. Iron-based powder mixtures were prepared by mixing this alloyed steel powder with 0.5% by mass of a graphite powder and various lubricants shown in Table 1. The mixing was effected with heat and by use of a high-speed mixer.

[0057] First, a die for compacting was preheated at each of the temperatures listed in Table 1. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the die and applied to the mold die surface by means of electrical charging. The die lubricant was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 1. The temperature measured on the mold surface was taken as a pressure compaction temperature.

[0058] Subsequently, the mold thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 x 10 x 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 1. A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants as shown in Table 1.

[0059] As a conventional example, a similar rectangular green compact (Green compact No. 38) was formed in the same manner as in Example 1 except that a die was not coated with a die lubricant for warm compaction with die.

[0060] After completion of the compaction, the force was measured which was required for the green compact to be

drawn from the mold.

[0061] With regard to each green compact thus formed, the density was determined by Archimedes' principle. The principle noted here denotes a method by which the density of a test specimen, each green compact in this case, is determined by measuring the volume of the product after immersion in ethyl alcohol. Additionally, visual inspection was made of the appearance of the green compact to find faults such as marring, breakage and the like. The green compact was centrally cut, embedded in resin and then abraded, followed by examination of voiding in section on a light microscope.

[0062] The drawing force, density, appearance and sectional structure of the green compact are tabulated in Table 1.

[0063] All the green compacts representing this invention show as low a drawing force after compaction as 20 MPa or below and as high a density as 7.4 Mg/m³ or above. Furthermore, these products are free of surface oxidation due to heating as well as faults such as marring, breakage and the like. The sectional structures are normal with the absence of coarse voids.

[0064] The comparative and conventional examples that fall outside the scope of the invention revealed a high drawing force exceeding 20 MPa, a low density of less than 7.35 Mg/m³, or coarse voids near to the sectional surface of the green compact.

[0065] Advantageously, the present invention can form a high-density green compact which exhibits superior appearance and sectional structure and low drawing force.

Example 2

[0066] The following six different powders were used as iron-based powders; namely (1) a partially alloyed steel powder a of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder, (2) a partially alloyed steel powder b of a Fe-2Ni-1Mo composition derived by diffusively bonding Ni and Mo to a pure atomized iron powder, (3) a prealloyed steel powder c of a Fe-3Cr-0.3Mo-0.3V composition derived by prealloying Cr, Mo and V, (4) a prealloyed steel powder d of a Fe-1Cr-0.3Mo-0.3V composition derived by prealloying Cr, Mo and V, (5) an atomized iron powder e, and (6) a reduced iron powder f. The atomized iron powder denotes an iron-based powder resulting from atomization of molten steel with high-pressure water, and the reduced iron powder denotes an iron-based powder resulting from reduction of iron oxide.

[0067] The partially alloyed steel powder a, partially alloyed steel powder b, prealloyed steel powder c, prealloyed steel powder d, atomized iron powder e and reduced iron powder f were each mixed with graphite in the contents shown in Table 3 and with the lubricants shown in Table 3, whereby iron-based powder mixtures were prepared. The mixing was effected with heat and by use of a high-speed mixer. In case of the atomized iron powder e and reduced iron powder f, 0.8% by mass of graphite and 2.0% by mass of a Cu powder were mixed. The content of graphite is by a mass ratio relative to the total amount of iron-based powder and graphite, or of iron-based powder, graphite and alloy powder.

[0068] First, a pressure compaction die was preheated at each of the temperatures listed in Table 3. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the mold and applied to the mold surface by means of electrical charging. The die lubricant for warm compaction with die lubrication was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 3. The temperature measured on the mold surface was taken as a pressure compaction temperature.

[0069] Secondly, the mold thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 × 10 × 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 3. A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants as shown in Table 3.

[0070] With regard to each iron-based green compact thus obtained, the density was determined by Archimedes' principle as in Example 1.

[0071] Subsequently, the iron-based green compact was sintered in a N₂-10%H₂ atmosphere and at 1,130 C for 20 minutes, whereby an iron-based sintered compact was formed. The density of the sintered compact was determined by Archimedes' principle. This product was then machined to obtain a sample in the shape of a small round rod dimensioned to be 5 mm in parallel plane diameter and 15 mm in length. The sample used to measure tensile strength.

[0072] Similar rectangular green compacts were formed in the same manner as in Example 2 except that a die was not coated with a die lubricant for warm compaction with die. Each green compact was further sintered as in Example 2 to form an iron-based sintered compact which was taken as a conventional example.

[0073] The test results are tabulated in Table 3.

[0074] The present invention provides high density and great tensile strength in contrast to the conventional examples (Sintered compacts Nos. 2 to 12).

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Table 1-1

Crown sample No.	Die lubricants		Lubricants in powder-in-based mixture				Composite conditions				Oven experiments		Results	
	Lubricant of higher melting point than competing temperature		Lubricant of lower melting point than competing temperature		Lubricant of lower melting point than competing temperature		Die preheating temperature		Composite conditions		Apparatus			
	Crown* sample No.%	Kind (melting point)	Crown* sample%	Kind (melting point)	Crown* sample%	Kind (melting point); Content by mass%**	Crown* sample%	Kind (melting point); Content by mass%**	Crown* sample%	Holding temperature of die and lubricant mixture	Ejection force N/m²	Density kg/m³	Saturation atmosphere	
1	5	A1(230°C)	95	A1(180 to 195°C)	0.050	A3(200°C)0.03	30	A1(140 to 195°C); Content by mass%**	20	210	150	160	17	good
2	5	A1(216°C)	95	A1(180 to 195°C)	0.200	A3(200°C)0.10	75	A1(140 to 195°C); Content by mass%**	25	210	150	160	18	good
3	5	B1(240°C)	95	B1(16°C)	0.310	A3(215°C)0.20	17	C1(170°C)0.10	43	220	155	170	18	bad
4	18	B2(240°C)	90	A1(17°C)	0.150	A1(215°C)0.10	35	A3(170°C)0.10	67	180	130	130	20	good
5	18	E1(16°C)	90	D1(18°C)	0.020	A1(180 to 195°C)	30	A1(180 to 195°C); Content by mass%**	50	210	145	160	20	good
6	18	F1(200°C)	90	D1(17°C)	0.200	A1(180 to 195°C)	60	C1(170°C)0.10	40	200	135	150	18	bad
7	18	F1(200 to 230°C)	90	D1(17°C)	0.400	A3(200°C)0.10	35	C1(170°C)0.10 A1(180 to 195°C)0.10	73	210	145	155	11	good
8	20	G1(>200°C)	60	C1(16°C)	0.150	A3(230°C)0.100	67	A1(170°C)0.025 A1(140 to 195°C)0.025	13	200	140	155	11	good
9	20	G1(>200°C)	80	A1(27°C)	0.050	A3(210°C)0.020	40	A3(170°C)0.015 C1(170°C)0.015	60	200	135	150	14	good
10	23	G1(>200°C)	75	A1(14 to 195°C)	0.200	A3(170°C)0.100	30	A1(180 to 195°C)0.25 A1(180 to 195°C)0.25 C1(170°C)0.15	50	210	150	160	11	good

* Content ratio to total volume lubricant.

** Total content of lubricants in powder-in-based mixture.

Note: Cross-refer to Table 2 as to lubricant symbols.

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Table 1-2

Grease compus. No.	Base lubricant	Lubricant properties based on mineral oil		Competitor conditions		Grease properties		Remarks									
		Lubricant of higher melting point than competing temperature	Lubricant of lower melting point than competing temperature	Oil preheating temperature	Compaction temperature for powdered iron-based matrix	Density	Appearance										
		Cold** max.%	Kind (melting point)	Kind (melting point) max.*** max.***	Cold*** max.%	°C	KPa	Mg/m ³									
11	25	H1(>200°C)	71	B1(146°C)	0.350	A4(216°C)0.100	29	A1(146°C ISCC 0.10 CH47C 0.100 ISCC 0.10 AS107C 0.075	71	200	145	12	7.41	good	good	Invention	
12	25	H2(>200°C)	73	C1(147°C)	0.150	A4(216°C)0.100	67	A1(146°C ISCC 0.10 CH47C 0.100 ISCC 0.10 AS107C 0.075	33	210	155	160	14	7.42	good	good	Invention
13	30	A3(230°C)	70	A2(137°C)	0.400	A4(216°C)0.100	25	A1(146°C ISCC 0.10 CH47C 0.100 ISCC 0.10 AS107C 0.075	75	180	135	130	12	7.40	good	good	Invention
14	30	A3(230°C)	70	A2(137°C)	0.050	A4(216°C)0.100	70	A1(146°C ISCC 0.010 AS107C 0.005 CH47C 0.015	30	185	125	135	15	7.43	good	good	Invention
15	30	A3(230°C)	70	C2(100°C)	0.400	A4(216°C)0.100	75	A1(146°C ISCC 0.10 AS107C 0.005 CH47C 0.010	23	185	120	135	13	7.43	good	good	Invention
16	33	A3(230°C)	63	A2(137°C)	0.400	A4(216°C)0.100	30	A1(146°C ISCC 0.10 CH47C 0.100 AS107C 0.005	50	190	130	140	13	7.40	good	good	Invention
17	35	A3(116°C)	65	D2(<177°C)	0.050	A3(230°C)0.025	50	A1(146°C ISCC 0.025 CH47C 0.025 AS107C 0.025	50	190	130	140	18	7.43	good	good	Invention
18	33	A3(230°C)	63	A1(137°C)	0.200	A1(146°C)0.100	75	A1(146°C ISCC 0.10 CH47C 0.100 AS107C 0.005	25	205	140	155	15	7.40	good	good	Invention
19	40	A3(230°C)	60	B1(165°C)	0.310	A3(230°C)0.100	57	C1(147°C)0.150	43	200	135	130	17	7.42	good	good	Invention
20	40	A4(216°C)20	60	C1(147°C)	0.150	A4(216°C)0.040	27	A1(146°C ISCC 0.10 CH47C 0.100 AS107C 0.020	73	200	135	130	17	7.42	good	good	Invention

*) continual cold to hot mineral lubricant

**) kind (number of lubricants in powdered iron-based mixture)

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Table 1-3

Green composi- tion No.	Distortions		Lubricants in powdered iron-based steels		Competing conditions		Green compac- tivity		Remarks							
	Lubricant of lower melting point than competing temperature	Lubricant of higher melting point than competing temperature	Lubricant of higher melting point than competing temperature	Lubricant of lower melting point than competing temperature	Kind (cooling point): Content (%) of metal(s)*	Content (%) of metal(s)*	Die penetration temperature for powdered iron-based steels	Ejection force N/mm ²	Density kg/m ³	Appearance	Structural structure					
21	40	Al(216°C) Et(246°C)	60	Bi(146°C)	0.010	Al(215°C)0.025	30	Al(188 to 155°C)0.01	50	205	140	135	16	7.42	good	good
22	40	Al(16°C) Et(16°C)	60	Bi(147°C)	0.200	Al(160°C)0.120	50	Cl(177°C)0.0	40	200	135	130	16	7.42	good	good
23	40	Et(30°C) Al(16°C)	60	Bi(148°C)	0.310	Al(215°C)0.150	43	Al(188 to 155°C)0.00	57	205	145	135	14	7.40	good	good
24	45	Et(206°C) G(146°C)	55	Al(146 to 157°C)	0.150	Al(210°C)0.100	67	Al(188 to 155°C)0.01	33	210	150	130	17	7.44	good	good
25	50	Et(206°C) H(146°C)	50	Al(146°C)	0.010	Al(210°C)0.023	40	Cl(177°C)0.015	60	210	150	130	16	7.43	good	good
26	50	Al(206°C) Et(206°C)	50	Bi(146°C)	0.200	Al(210°C)0.100	50	Al(188 to 155°C)0.015	50	213	155	135	19	7.42	good	good
27	50	Al(16°C) Et(16°C)	50	Bi(146°C)	0.110	Al(210°C)0.100	29	Al(188 to 155°C)0.010	71	215	155	133	20	7.41	good	good
28	2	Et(290°C)	94	Bi(147°C)	0.010	Al(215°C)0.010	30	Al(188 to 155°C)0.005	50	220	160	170	19	7.46	good	good
29	2	Et(290°C)	96	Bi(146°C)	0.400	Al(215°C)0.100	23	Cl(177°C)0.02	75	220	160	170	17	7.39	good	good
30	10	Al(210°C)	90	Al(146 to 155°C)	-	-	-	-	-	210	145	140	15	7.31	good	good

content ratio to clinkosil lubricant:
1) total content of lubricant in powdered iron-based steels
*) content is powdered iron-based steels

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Table 1-4

Cetene content wt-% Name	Die lubricants		Lubricants in powdered iron-based mixtures		Lubricants in solidified iron-based mixtures that contain no softening temperature		Concreting conditions		Curing conditions		Remarks				
	Cetene ^a wt-%	Kild (rolling point)	Cetene ^b wt-%	Kild (rolling point)	Cetene ^c wt-%	Kild (rolling point); Cetene ^d wt-%	Cetene ^e wt-%	Die protective temperature °C	Heatline distance or probe-head distance mm	Die protective temperature °C	Heatline distance or probe-head distance mm	Extraction force N/mm ^f	Density kg/m ³	Sediment status	
31 10	AJ(20°C)	90	B(146°C)	0.90	AJ(20°C)0.100	50	AJ(177°C)0.300	50	190	123	110	29	7.33	united	good
32 2	AJ(20°C)	94	C(147°C)	0.10	AJ(20°C)0.143	95	C(147°C)0.075	5	190	125	140	31	7.34	united	good
33 15	AJ(20°C)	23	AZ(27°C)	0.10	AJ(20°C)0.070	20	AZ(177°C)0.120	10	110	113	110	17	7.40	void	void
34 0	-	100	A(148 to 151°C)	0.30	AJ(20°C)2.20	80	A(148 to 151°C)0.070	20	210	159	150	25	7.42	united	good
35 100	A(131°C) A(148 to 151°C)	0	-	0.200	AJ(20°C)0.100	50	AJ(177°C)0.100	10	190	125	140	10	7.27	good	good
36 100	A(220°C) A(148 to 151°C)	0	-	0.300	AJ(20°C)2.22	75	A(148 to 151°C)0.075	25	100	139	60	25	7.27	good	good
37 0	-	100	A(148 to 151°C)	0.200	AJ(20°C)0.23	75	A(148 to 151°C)0.075	25	170	210	220	29	7.41	oxidized	good
38	-	-	-	0.000	AJ(20°C)0.420	70	AJ(148 to 151°C)0.100	30	220	160	170	18	7.35	united	good

^a) content ratio to total mineral lubricant
^b) total content of lubricant in powdered iron-based mixture
^c) content to powdered iron-based mixture

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Table 2

Symbol	Kinds of lubricants	Symbols	Kinds of lubricants		
			E1	E2	E3
A1	Calcium stearate	Metallic soap	Polymer	Thermoplastic resin	
A2	Zinc stearate		Polyamide (nylon 6)		
A3	Lithium stearate		Polyethylene		
A4	Lithium hydrocarboxylate		Polyisobutylene		
B1	Silicate calcium low-density polyethylene	F2	Polyamide elastomer		
C1	Ethylene bis-stearoate	Amino wax	G1	Graphite	Inorganic lamellar lubricant
C2	Monomide stearate		G2	MoS ₂	
D1	Ethylene bis-stearamide- polyethylene esterific	Esteric acid	G3	Carbox fluoride	Organic lamellar lubricant
D2	Ethylene bis-stearamide- zinc stearate esterific		H1	Methacrylate acid acidic (MCA)	
D3	Ethylen bis-stearamide- calcium stearate esterific		E2	N-Allyl isopropene- olefin	

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Table 3

Sintered Compact No.	Die lubrication		Kind of graphite powder in powder mixture mass-%	Lubrication is provided from-based mixtures				Compacting conditions				Remarks						
	Lubricant of higher melting point than compacting temperature			Lubricant content** mass-%	Lubricant of lower melting point than compacting temperature		Die melting temperature for powdered graphite mass-%	Die melting temperature for powdered graphite mass-%	Die melting temperature for powdered graphite mass-%	Die melting temperature for powdered graphite mass-%								
	Content* mass-%	Kin'd. (melting point)			Kin'd. (melting point) Catalytically modified graphite	Kin'd. (melting point) graphite												
2-1	75	A1(20°C)	25	A1(48 to 155°C)	4	0.6	0.20	A1(20°C)0.45	75	A1(48 to 155°C)0.05	25	210	150	160	7.42	240	\$20	Invention
2-2	-	-	-	-	4	0.6	0.20	A1(20°C)0.60	73	A1(48 to 155°C)0.20	25	210	150	160	7.22	231	740	Conventional Example
2-3	75	A1(20°C)	25	A1(48 to 155°C)	6	0.6	0.20	A1(20°C)0.15	73	A1(48 to 155°C)0.05	25	210	150	160	7.42	243	710	Invention
2-4	-	-	-	-	6	0.6	0.20	A1(20°C)0.60	73	A1(48 to 155°C)0.20	25	210	150	160	7.33	234	640	Conventional Example
2-5	75	A1(20°C)	25	A1(27°C)	6	0.9	0.20	A1(48 to 155°C)0.05	50	A1(13°C)0.01	50	165	120	133	7.23	222	\$10	Invention
2-6	-	-	-	-	6	0.9	0.20	A1(48 to 155°C)0.20	50	A1(13°C)0.49	50	165	120	133	7.13	712	770	Conventional Example
2-7	75	A1(20°C)	25	A1(27°C)	4	0.9	0.20	A1(48 to 155°C)0.05	50	A1(13°C)0.10	50	165	120	133	7.23	233	650	Invention
2-8	-	-	-	-	4	0.9	0.20	A1(48 to 155°C)0.20	50	A1(13°C)0.40	50	165	120	133	7.13	713	760	Conventional Example
2-9	75	A1(20°C)	25	C2(100°C)	*	0.8	0.20	A1(20°C)0.10	50	A1(27°C)0.03	30	170	115	130	7.25	223	620	Invention
2-10	-	-	-	-	*	0.8	0.20	A1(20°C)0.40	75	A1(27°C)0.10	25	170	115	130	7.27	714	530	Conventional Example
2-11	75	A1(20°C)	25	C2(100°C)	f	0.8	0.20	A1(20°C)0.10	50	A1(27°C)0.03	30	170	115	130	7.25	714	650	Invention
2-12	-	-	-	-	f	0.8	0.20	A1(20°C)0.60	75	A1(27°C)0.10	25	170	115	130	7.16	705	590	Conventional Example
2-13	75	A1(20°C)	25	A1(48 to 155°C)	*	0.8	0.20	A1(20°C)0.13	75	A1(48 to 155°C)0.05	25	210	150	160	7.40	739	620	Invention

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Standard Impact No.	One substance		Lubricants in powdered iron-based mixtures		Compacting conditions		Green consistency		Standard Compressive Strength		Remarks
	Kind of iron-based powder	Grinding coarse or fine & hard powder mixture earlier	Lubricant kind earlier	Lubricant of higher viscosity or than compacting temperature	Kind (coating point); Content by mass %	Kind (coating point); Content by mass %	Heating temperature for powdered mixture	Heating temperature for powdered mixture	Density	MPa	
2-14	75	A1(20°C)	25	A1(148 to 157°C)	1.1	0.20	A1(20°C)0.15	73	1.60	736	730
2-15	75	A1(20°C)	25	A1(148 to 157°C)	1.3	0.20	A1(20°C)0.15	73	1.60	732	731

* Content also to total amount lubricant
** Lubricant is a hard, firm, brittle mixture
*** Content of powder is 10% of the total weight of the mixture
**** Content of powder is 15% of the total weight of the mixture
Note: Corresponding to Table 4 to the lubricant properties.

Example 3

[0075] A partially alloyed steel powder of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder was used as an iron-based powder. Iron-based powder mixtures were prepared by mixing this alloyed steel powder with 0.2% by mass of a graphite powder and various lubricants shown in Table 3. The mixing was effected with heat and by use of a high-speed mixer.

[0076] First, a pressure compaction die was preheated at each of the temperatures listed in Table 4. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the die and applied to the die surface by means of electrical charging. The die lubricant for warm compaction with die lubrication was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 4. The temperature measured on the die surface was taken as a pressure compaction temperature.

[0077] Subsequently, the mold thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 x 10 x 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 4. A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants as shown in Table 4.

[0078] As a conventional example, a similar rectangular green compact (Green compact No. 38) was formed in the same manner as in Example 4 except that a die was not coated with a die lubricant for warm compaction with die.

[0079] After completion of the compaction, the ejection force was measured.

[0080] With regard to each of the resultant green compacts, the density was determined by Archimedes' principle. Visual inspection was then made of the appearance of the green compact to find faults such as marring, breakage and the like. The green compact was centrally cut, embedded in resin and then abraded, followed by examination of voiding in section on a light microscope.

[0081] The drawing force, density, appearance and sectional structure of the green compact are tabulated in Table 4.

[0082] All the green compacts according to this invention show as low a drawing force after compaction as 20 MPa or below and as high a density as 7.43 Mg/m³ or above. In addition, each such product causes neither surface oxidation resulting from heating nor faults such as marring, breakage and the like. The sectional structure is normal with the absence of coarse voids.

[0083] The comparative and conventional examples that depart from the scope of the invention suffered a high drawing force exceeding 20 MPa, a low density of less than 7.39 Mg/m³, or coarse voids near to the sectional surface of the green compact.

[0084] The present invention is highly advantageous in that a high-density green compact is obtainable with superior appearance and sectional structure as well as low drawing force.

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Table 4-1

Grease compat- ibility No.	Diluents		Lubricants is provided time-based mixture		Compatibility conditions		Other characteris-			Remarks	
	Lubricant of higher melting point than competing temperature		Lubricant of lower melting point than competing temperature		Die prüfung temperatur bei verschieden Zeitablauf		Eigene Fähigkeit zur Abtrennung der verschieden Zeitablauf				
	Coldast* value %	Kin* (softening point)	Coldast* value %	Kin* (softening point)	Kind** Cement point: Cement by DIN 51814***	Kind** Cement point: Cement by DIN 51815***	Temperatur C	Temperatur C	Dichte kg/m³	Appearan- ce	
1	2	A3(210°C)	98	A1(148 to 155°C)	0.050	A1(148 to 155°C)	50	A1(148 to 155°C) 0.075	150	150	good
2	3	A1(216°C)	95	A2(127°C)	0.150	A1(216°C) 0.100	67	A1(148 to 155°C) 0.050	150	150	good
3	5	E1(240°C)	95	B1(146°C)	0.150	A1(220°C) 0.200	57	C1(147°C) 0.150	43	160	good
4	10	E1(210°C)	90	A1(210°C) 0.0	0.150	A1(210°C) 0.0	31	A2(132°C) 0.08	67	160	120
5	10	E1(246°C)	90	D1(147°C)	0.050	A1(215°C) 0.25	52	A1(148 to 155°C) 0.025	150	150	good
6	15	F1(200°C)	65	D1(127°C)	0.150	A1(165°C) 1.10	50	C1(147°C) 0.070	40	150	135
7	20	F1(110 to 210°C)	60	D1(147°C)	0.050	A1(210°C) 1.00	23	C1(147°C) 0.15	73	150	145
8	10	G1(210°C)	90	C1(117°C)	0.150	A1(210°C) 1.00	67	A1(117°C) 0.12	33	165	140
9	20	G1(210°C)	60	A1(177°C)	0.200	A1(210°C) 1.00	40	A2(227°C) 0.150	60	150	135
10	20	G1(210°C)	30	A1(148 to 155°C)	0.250	A1(210°C) 1.00	50	A1(148 to 155°C) 0.125	50	150	150

*
coldast value to solid cement/lubricant
** coldast value of lubricant in point-based mixture
*** content in powdered iron-based mixture
Note: Compare to Table 2 as to the lubricant symbols.

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Table 4.2

Green compact No.	Dimensions	Lubricants in powdered iron-based mixture				Comparative conditions				Green compacts			Remarks
		Lubricant of higher melting point than compacting temperature	Lubricant of higher melting point than compacting temperature	Lubricant of lower melting point than compacting temperature	Die preheating temperature	Die preheating temperature	Compacting temperature	Density	Apperance	Stoichiometric structure			
Content* metal-%	Kin. (melting point)	Content* metal-%	Content* metal-%	Content* metal-%	Temp. °C	Temp. °C	MPa	Mg/m ²					
11 25	H1(200°C)	75	B1(140°C)	Content* metal-%	AK16C10/10	50	A1(140°C)10/10 Cu(120°C)10/10	39	190	145	1.9	7.45	good
12 50	H2(200°C)	50	C1(147°C)	67	AK16C10/10	67	A1(140°C)10/10 Cu(120°C)10/10	33	190	135	1.6	7.46	good
13 30	A3(230°C)	70	A2(137°C)	25	AK16C10/10	840	CH(120°C)10/10 A1(120°C)10/10	75	160	120	1.0	7.45	good
14 60	A3(230°C)	40	A2(137°C)	67	AK16C10/10	0.50	AK16C10/10 A1(120°C)10/10	33	165	135	1.6	7.49	good
15 30	A3(230°C)	70	C2(100°C)	0.60	AK16C10/10 A1(120°C)10/10 A1(110°C)10/10 Cu(120°C)10/10	73	AK16C10/10 A1(120°C)10/10 Cu(120°C)10/10	25	160	120	1.0	7.46	good
16 35	A3(230°C)	65	A2(137°C)	0.200	AK16C10/10 A1(120°C)10/10	50	AK16C10/10 Cu(120°C)10/10	50	170	120	1.0	7.45	good
17 35	A4(160°C)	65	I2(147°C)	0.130	AK16C10/15	65	AK16C10/15	17	170	130	1.1	7.47	good
18 35	A4(160°C)	65	A1(150°C)15	0.100	AK16C10/15	73	A1(140°C)10/10	23	190	140	1.5	7.44	good
19 60	A4(160°C)	40	B1(146°C)	0.130	AK16C10/20	57	CH(140°C)10/10	43	180	135	1.5	7.46	good
20 40	A4(160°C)	60	C1(147°C)	0.130	AK16C10/10 E1(200°C)2	27	A1(137°C)10/10	73	180	135	1.5	7.46	good

* Content ratio to total amount of lubricants

** Lubricant of lubricant in powdered iron-based mixture

*** Content in powder iron-based mixture

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Table 4-3

Green Catalyzed No.	Catalyst Type Molar %	Dissolution		Lubricity by penetration test-based mixture		Construction conditions		Green conditions		Remarks				
		Lubricity at higher melting point than compaction temperature	Lubricity at lower melting point than compaction temperature (Δ 20°C)	Lubricity at higher melting point than compaction temperature	Lubricity at lower melting point than compaction temperature	Heating temperature (or provided in table)	Compacting temperature	Effecting force NPa	Density kg/dm^3					
21	40	Al(OEt) ₃ Et ₂ AlCl ₂	60	D1(<146°C)	0.150	Al(146 to 150°C)/0.050	67	190	140	155	13	7.46	good	good
22	40	Al(OEt) ₃ Et ₂ AlCl ₂	60	D1(<137°C)	0.120	Al(146 to 150°C)/0.010	40	180	135	150	16	7.43	good	good
23	30	Al(OEt) ₃ Et ₂ AlCl ₂	10	D1(<146°C)	0.210	Al(146 to 150°C)/0.100	43	190	145	155	14	7.44	good	good
24	30	Et ₂ Al(OEt) ₂ Et ₂ AlCl ₂	30	A1(146 to 151°C)	0.150	Al(146 to 150°C)/0.035	67	190	150	160	17	7.48	good	good
25	60	Et ₂ Al(OEt) ₂ Hf(2-20°C)O ₂	40	A1(137°C)	0.050	Al(146 to 150°C)/0.010	40	180	140	150	16	7.47	good	good
26	70	Al(OEt) ₃ Et ₂ AlCl ₂	30	B1(146°C)	0.200	Al(146 to 150°C)/0.100	50	190	155	165	15	7.45	good	good
27	40	Al(OEt) ₃ Et ₂ AlCl ₂ Et ₂ AlCl ₂	20	D1(<146°C)	0.210	Al(146 to 150°C)/0.010	71	190	155	165	19	7.45	good	good
28	2	Et ₂ Al(OEt) ₂	98	D1(<137°C)	0.200	Al(146 to 150°C)/0.100	50	210	160	170	19	7.49	good	good
29	2	Et ₂ Al(OEt) ₂	98	D1(<146°C)	0.400	Al(146 to 150°C)/0.100	25	210	160	170	17	7.43	good	good
30	10	Al(OEt) ₃	90	A1(146 to 151°C)	-	-	-	190	145	160	35	7.35	good	good

¹⁾ Cetane ratio of total amount (lubricant
base oil and catalyst) to total amount in penetration test-based mixture
^{**})

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Table 4-4

Green Composi- tions No.	Oil lubricating		Lubricants in powdered iron-based matrices		Combustion conditions		Chemical properties		Remarks								
	Lubricant of higher melting point than corresponding temperature	Lubricant at lower melting point than corresponding temperature	Lubricant obtained + 5%	Lubricant of higher melting point than corresponding temperature	Lubricant using temperature	Die penetration resistance method Crash, mm ²	Crash ^a , mm ²	Ejection force N/mm ²									
21	10	Al(210°C)	90	B1(165°C)	0.600	Al(120°C)0.300	10	Al(177°C)0.100	50	150	124	150	29	7.16	marked	good	Cooperative Example
22	5	Al(210°C)	95	C1(167°C)	0.250	Al(120°C)0.143	55	C1(149°C)0.0073	2	180	123	150	21	7.18	marked	good	Cooperative Example
23	20	Al(210°C)	10	A2(137°C)	0.150	Al(140°C)0.010	20	Cr(180°C)0.120	80	160	115	130	23	7.42	good	yield	Cooperative Example
24	9	-	100	A1(148 to 155°C)	0.200	Al(120°C)0.200	67	Al(148 to 155°C)0.10	33	190	130	160	25	7.44	marked	good	Cooperative Example
25	120	Al(210°C) Al(198 to 195°C)	0	-	0.200	Al(120°C)0.100	10	Al(177°C)0.100	50	170	125	140	30	7.31	good	good	Cooperative Example
26	120	Al(200°C) Al(198 to 195°C)	0	-	0.300	Al(120°C)0.223 Al(148 to 155°C)0.075	100	-	2	160	60	70	25	7.31	good	good	Cooperative Example
27	2	-	100	Al(148 to 155°C) Al(210°C)25	0.300	Al(120°C)0.225 Al(210°C)25	75	Al(148 to 155°C)0.073	25	250	210	210	30	7.47	marked	good	Cooperative Example
28	-	-	-	-	0.600	Al(210°C)0.410	70	Al(148 to 155°C)0.10	30	200	160	170	28	7.38	marked	good	Conventional Example

^a) contact ratio to total amount lubricant
 *) total amount of lubricants in powdered iron-based matrix
 **) content in powdered iron-based matrix

Example 4

[0085] The following two different powders were used as iron-based powders; namely (1) a partially alloyed steel powder a of a Fe-4Ni-0.5Mo-1.5Cu composition derived by diffusively bonding Ni, Mo and Cu to a pure atomized iron powder, and (2) a prealloyed steel powder b of a Fe-3Cr-0.3Mo-0.3V composition derived by prealloying Cr, Mo and V.

[0086] The partially alloyed steel powder a, and prealloyed steel powder b were mixed with graphite in the contents shown in Table 5 and the lubricants shown in Table 5, whereby iron-based powder mixtures were prepared. The mixing was effected with heat and by use of a high-speed mixer. The content of graphite is by a mass ratio relative to the total amount of the iron-based powder mixture.

[0087] First, a die was preheated at each of the temperatures listed in Table 5. A die lubricant for warm compaction with die electrically charged by a die lubricating system (manufactured by Gasbarre Co.) was jetted into the die and applied to the die surface by means of electrical charging. The die lubricant for warm compaction with die lubrication was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants shown in Table 2, and then by formulating both lubricants as shown in Table 5. The temperature measured on the mold surface was taken as a pressure compaction temperature.

[0088] Secondly, the die thus treated was filled with a heated iron-based powder mixture, followed by pressure compaction, whereby a rectangular green compact with a size of 10 x 10 x 55 mm was produced. The pressure loading was 686 MPa, and other pressure compaction conditions were as listed in Table 5.

[0089] A powder compaction lubricant contained in the iron-based powder mixture was prepared by choosing a lower-melting lubricant and a higher-melting lubricant from among the lubricants listed in Table 2, and then by formulating both lubricants as shown in Table 5.

[0090] With regard to each iron-based green compact thus obtained, the density was determined by Archimedes' principle as in Example 1.

[0091] Subsequently, the iron-based green compact was sintered in a N₂-10%H₂ atmosphere and at 1,130 C for 20 minutes, whereby an iron-based sintered compact was formed. The density of the resultant sintered compact was determined by Archimedes' principle. The test results are tabulated in Table 5. The examples of the invention provides high densities.

[0092] As stated above, the present invention is significantly advantageous in that a high-density green compact can be produced with superior appearance and sectional structure and by single compaction. Drawing of the product from the associated mold is possible at a low force with a prolonged lifetime of the die. Also notably, a high-density sintered compact is easy to produce.

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Table 5

Sample Number and Design No.	Disinfectants		Lubricants (provided from-based mixtures)		Competing emulsifiers		Gums contents	Silicon contents	Density	Remarks							
	Lubricant of higher melting point than competing emulsifier	Kind of lubricant used and percentage	Lubricant content mass%	Lubricant of higher melting point than competing emulsifier	Kind (existing point): Content mass% of existing point	Kind (existing point): Content mass% of existing point											
2-1	73	A1(20°C)	25	A1(48 to 155°C)	4	0.15	0.15	A1(20°C)0.1	67	A1(48 to 155°C)0.075 A1(27°C)0.025	73	150	160	7.49	7.47	Invention	
2-2	75	A1(20°C)	25	A1(48 to 155°C)	6	0.30	0.15	A1(20°C)0.1	67	A1(48 to 155°C)0.075 A1(27°C)0.025	30	150	120	6.69	7.47	Invention	
2-3	75	A1(20°C)	25	A1(48 to 155°C)	4	0.45	0.15	A1(20°C)0.1	67	A1(48 to 155°C)0.075 A1(27°C)0.025	30	150	130	6.60	7.43	Invention	
2-4	75	A1(20°C)	25	A1(48 to 155°C)	6	0.35	0.15	A1(20°C)0.1	67	A1(48 to 155°C)0.075 A1(27°C)0.025	30	150	120	6.60	7.39	Comparative Example	
2-5	75	A1(20°C)	25	A1(127°C)	6	0.15	0.20	A1(48 to 155°C)0.03 C1(47°C)0.05	50	A1(127°C)0.10	50	165	120	135	7.34	7.34	Invention
2-6	75	A1(20°C)	25	A1(127°C)	6	0.20	0.20	A1(48 to 155°C)0.03 C1(47°C)0.05	50	A1(127°C)0.10	50	165	120	135	7.32	7.32	Invention
2-7	75	A1(20°C)	25	A1(127°C)	6	0.45	0.20	A1(48 to 155°C)0.03 C1(47°C)0.05	50	A1(127°C)0.10	50	165	120	135	7.30	7.29	Invention
2-8	75	A1(20°C)	25	A1(127°C)	6	0.15	0.20	A1(48 to 155°C)0.03 C1(47°C)0.05	50	A1(127°C)0.10	50	165	120	135	7.25	7.24	Comparative Example

^a) content ratio to total amount lubricant

^b) total content of lubricants in provided from-based mixture

Note: Crossrefer to Table 2 to the lubricant symbols.

Claims

1. A die lubricant for warm compaction with die lubrication, comprising a mixture of a lubricant having a higher melting point than a preset compaction temperature, and a lower melting point than the compaction temperature, the die lubricant for warm compaction with die lubrication being applicable to the surface of a preheated die by means of electrical charging when a powdered material is compacted by pressure compaction.
2. A die lubricant for warm compaction with die lubrication, comprising a lubricant having a higher melting point than a preset compaction temperature and in a content from 0.5 to 80% by mass, and a lower melting point than the compaction temperature and as the balance, the die lubricant for warm compaction with die lubrication being applicable to the surface of a preheated die by means of electrical charging when a powdered material is compacted by pressure compaction.
3. The die lubricant for warm compaction with die lubrication according to claim 2, wherein the higher-melting lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure.
4. The die lubricant for warm compaction with die lubrication according to claim 2, wherein the lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.
5. An iron-based powder mixture for warm compaction with die lubrication, comprising an iron-based powder and a powder compaction lubricant, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.
6. An iron-based powder mixture for warm compaction with die lubrication, comprising an iron-based powder, a powder compaction lubricant and a graphite powder, wherein the powder compaction lubricant comprises a lubricant having a lower melting point than a preset compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance, and the content of the graphite powder is less than 0.5% by mass based on the total amount of the iron-based powder mixture.
7. The iron-based powder mixture for warm compaction with die lubrication, wherein the content of the powder compaction lubricant is in the range from 0.05 to 0.40% by mass.
8. A process for the production of a high-density iron-based green compact, comprising the steps of: preheating a die at a selected temperature; applying a die lubricant for warm compaction with die lubrication to a surface of the die by means of electrical charging; filling a heated iron-based powder mixture in the die; and then subjecting the powder mixture to pressure compaction at a preset compaction temperature, wherein the lubricant for warm compaction with die lubrication comprises a lubricant having a higher melting point than the compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance; and the iron-based powder mixture comprises an iron-based powder and a powder compaction lubricant, the powder compaction lubricant comprising a lubricant comprising a lubricant having a lower melting point than the compaction temperature and in a content from 10 to 75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance.
9. A process for the production of a high-density iron-based green compact, comprising the steps of: preheating a die at a selected temperature; applying a lubricant for warm compaction with die lubrication to the surface of the die by means of electrical charging; filling a heated iron-based powder mixture into the die; and then subjecting the powder mixture to pressure compaction at a present compaction temperature, wherein the die lubricant for warm compaction with die lubrication comprises a lubricant having a higher melting point than the compaction temperature and in a content from 0.5 to 80% by mass, and a lubricant having a lower melting point than the compaction temperature and as the balance; and the iron-based powder mixture comprises an iron-based powder, a powder compaction lubricant and a graphite powder, the powder compaction lubricant comprising a lubricant comprising a lubricant having a lower melting point than the compaction temperature and in a content from 10 to

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75% by mass based on the total amount of the powder compaction lubricant, and a lubricant having a higher melting point than the compaction temperature and as the balance; and a graphite powder being added in a content less than 0.5% by mass based on the total amount of the iron-based powder mixture.

- 5 10. The process according to one of claims 9 and 10, wherein the higher-melting die lubricant is at least one selected from the group consisting of metallic soap, thermoplastic resin, thermoplastic elastomer, and an organic or inorganic lubricant having a lamellar crystal structure.
- 10 11. The process according to one of claims 8 and 9, wherein the lower-melting lubricant is at least one selected from the group consisting of metallic soap, amide wax, polyethylene, and an eutectic mixture of at least two members thereof.
- 15 12. The process according to any one of claims 8 to 11, wherein the lubricant for warm compaction lubrication is added in a content from 0.05 to 0.40% by mass.
13. A process for the production of a high-density iron-based sintered compact, comprising the step of sintering the high-density iron-based green compact produced by the process according to any one of claims 8 to 12, thereby forming a sintered compact.

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INTERNATIONAL SEARCH REPORT		International application No. PCT/JP00/05089
A. CLASSIFICATION OF SUBJECT MATTER Int.Cl' B22F 1/00, 3/02, 3/035, C22C 33/02		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) Int.Cl' B22F 1/00, 3/02, 3/035, C22C 33/02		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Jitsuyo Shinan Koho 1926-1996 Toroku Jitsuyo Shinan Koho 1994-2000 Kokai Jitsuyo Shinan Koho 1971-2000 Jitsuyo Shinan Toroku Koho 1996-2000		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP, 10-280005, A (Kawasaki Steel Corporation), 20 October, 1998 (20.10.98) (Family: none)	1-13
Y	JP, 11-193404, A (Hitachi Powdered Metals co., Ltd.), 21 July, 1999 (21.07.99) (Family: none)	1-13
Y	US, 5744433, A (Storstrom et al.), 28 April, 1998 (28.04.98) & JP, 10-501270, A (Hoganasu Akuchiboragetto), 03 February, 1998 (03.02.98)	1-13
Y	JP, 9-71801, A (Kawasaki Steel Corporation), 18 March, 1997 (18.03.97) (Family: none)	1-13
Y	US, 5682591, A (Inculet et al.), 28 October, 1997 (28.10.97) & JP, 8-100203, A	1-13
<input type="checkbox"/> Further documents are listed in the continuation of Box C.		<input type="checkbox"/> See patent family annex.
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Date of the actual completion of the international search 24 October, 2000 (24.10.00)		Date of mailing of the international search report 07 November, 2000 (07.11.00)
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